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Formation of a nano-crystalline layer on the surface of glass fibres

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The Fe-Mg-Ca-aluminosilicate glass (FMCAS glass) starts to crystallize at an onset temperature (T_{onset}) well above its glass transition temperature (T_g), when it is upscanned at 20 K/min in a calorimeter. Such crystallization is here referred to as high temperature (HT) crystallization. The details (e.g. extent, rate and T_{onset}) of the HT crystallization depend on chemical composition, heating atmosphere, and heating rate, of the glass. This is already known in literature. What is less well known is that the FMCAS glass also crystallizes around T_g far below the T_{onset} of the HT crystallization, if the glass contains certain amount of iron, and has sufficiently high Fe^{2+}/Fe^{3+} ratio, and is heat-treated in oxidizing atmosphere. This crystallization process is featured by the formation of nano-crystals (e.g. periclase) on the glass surface, which is driven by two simultaneous processes: oxidation of Fe^{2+} and outward cationic diffusion. Such crystallization is here referred to as low temperature (LT) crystallization. The LT crystallization is not detectable by calorimetry. Therefore, other techniques such as secondary neutral mass spectrometry, atomic force microscopy and X-ray diffraction have been employed to identify the type, the structure, and the size of crystals formed upon LT heat-treatment. Differential scanning calorimetry has been used to measure thermal response to the HT crystallization and oxidation of Fe^{2+}. Mössbauer spectroscopy has been used to determine the Fe^{2+}/Fe^{3+} ratio. Here we present results of LT crystallization of the FMCAS glass fibres, and describe the mechanisms of both LT and HT crystallization processes. The results show that the amount of the nano-crystals formed at lower temperatures has strong impact on the relative ratio of different crystalline phases occurring during the subsequent HT heating, and on the extent of HT crystallization. The nano-crystalline layer influences the physical and chemical properties of the FMCAS glass fibres.